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Analytical and Experimental
Investigations of the Fracture
Mechanisms of Controlled
Polyphase Alloys

February 28, 1963

Prepared under Navy, Bureau of Naval Weapons
Contract N600(19)59361

Quarterly Progress Report No. 1

Period: October 29, 1962 through January 29, 1963

REPORTED BY J. A. Ford
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This report applies to work
on Contract N600(19)59361

SUMMARY

The results of preliminary investigations on selected unidirectionally solidified binary eutectic alloys are reported in this quarterly progress report. These investigations have shown that the Cu-Cr and Al-Al_3Ni systems can be successfully unidirectionally solidified with only very occasional growth defects, bands, such that highly oriented microstructures result. Large ingots of the Al-$CuAl_2$ system have not been produced free of bands to date. However, several ingots with only slight or light bands have been obtained. The Al-Al_3Ni system has been chosen as an alternate for the Cr-$Cr_{23}C_6$ system, both rod-type eutectics, since the reinforcing rod or whisker in the latter system has been determined to be Cr rather than $Cr_{23}C_6$.

Mechanical testing of several unidirectionally solidified Cu-Cr ingots, produced at fast growth rates, indicate that failure occurs by a combined shear and tensile mode. Similar experiments on as-cast and unidirectionally solidified Al-Al_3Ni ingots which contain 11 volume percent of a fibrous Al_3Ni phase indicate that controlled solidification has lead to the production of a reinforced structure in which the load is successfully transferred to the fibers by the matrix. Several Cr whiskers, $< 1\mu$ diameter, have been tensile tested and have shown elastic strengths in excess of 1,000,000 psi and elastic moduli of 35 - 40,000,000 psi.

Analytical and Experimental Investigations of the Fracture Mechanisms of Controlled Polyphase Alloys

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Analytical and Experimental Investigations of the Fracture Mechanisms of Controlled Polyphase Alloys

INTRODUCTION

This is the first quarterly progress report for Contract No. N600(19)59361 entitled "Analytical and Experimental Investigations of the Fracture Mechanisms of Controlled Polyphase Alloys," covering the period from October 29, 1962 through January 29, 1963. The objective of this investigation is to determine the role of highly oriented fibrous and lamellar second-phase particles on the mechanical properties of selected eutectic alloys.

Considerable experience has been obtained at the Research Laboratories in the production of unidirectionally solidified binary eutectic alloys which have their phases aligned parallel to one another and to the direction of solidification. The microstructure of these eutectics may be either rods of one phase in a matrix of the other or alternating plates of each phase. Systems representative of each morphology were chosen for the present investigation, Cu-Cr and Cr-$Cr_{23}C_6$ as rod-type and Al-$CuAl_2$ as lamellar-type. However, preliminary investigations of the Cr-$Cr_{23}C_6$ system on this contract have shown that the phase relationships are not as previously reported in the literature by other investigators. The Al-Al_3Ni system has been chosen as an alternate for investigation under this contract since preliminary work performed under UAC Research Laboratories' sponsorship indicated that the microstructure consisted of 11 volume percent Al_3Ni rods in an Al matrix.

This report includes a brief description of the experimental procedures, results of preliminary tests, and work to be conducted during the next report period.

CONTROLLED SOLIDIFICATION PROCEDURE

Cu-Cr System

Master heats of eutectic composition using OFHC Cu (~ 99.85) and Cr (99.85+) were prepared in two ingot sizes, ½-in. dia and 1½-in. dia, by vacuum induction melting in a MgO crucible and casting into silica molds. These ingots were uni-

directionally solidified in vertical quartz crucibles using a high-frequency induction source and graphite susceptor and an argon atmosphere. Solidification rates were varied from 1.2 to 11.0 cm/hr for the ½-in. dia bars which are to be used to study the fracture mechanism as a function of growth rate, and were varied from 1.2 to 2.8 cm/hr for 1½-in. dia ingots which are to be used to study mechanical behavior as a function of Cr-fiber orientation.

Al-Al_3Ni System

A master heat of 5.7 weight percent Ni was prepared from 99.99% Al and Ni by vacuum induction melting in a Al_2O_3 crucible and casting in a SiO_2 mold. The ½-in. dia ingots were then unidirectionally solidified in vertical graphite crucibles using an induction heat source at rates from 1 to 3 cm/hr in an argon atmosphere. The microstructure of this system is illustrated in Fig. 1a and 1b, transverse and longitudinal sections, showing the Al_3Ni fibers in the Al matrix.

Al-$CuAl_2$ System

Master heats of two sizes, rectangular 3/8-in. x 1½-in. x 5½-in. long and cylindrical 1½-in. dia x 5½-in. long, were prepared from pure (99.9965 Al and 99.999 Cu) and impure (99.99 Al and 99.85 Cu) starting material by vacuum induction melting in Al_2O_3 crucibles and casting in SiO_2 molds. Several 1½-in. dia x 10-in. long ingots were prepared by induction melting in graphite crucibles under an argon atmosphere using the same purity materials. The best solidification rate, as determined from previous work, appears to be 2 cm/hr.

The rectangular ingots were unidirectionally solidified in a horizontal graphite boat using a resistance heater. Attainment of good controlled lamellar structure was difficult using this geometry since heat flow is very unsymmetrical. Numerous attempts were made, including water cooling and vertical solidification, to produce planar liquid-solid interfaces which would result in highly controlled microstructures. The use of a more symmetric geometry to produce uniform heat flow, the cylindrical 1½-in. x 5½-in. ingots, resulted in the production of very large-grained controlled lamellar microstructure. The size of the grains produced by this technique is illustrated in Fig. 2.

Banding, illustrated in Fig. 3, is another solidification defect which will require further unidirectic... solidification experiments. The exact origin of banding is not understood; however, it is felt that perturbations in the growth rate and thermal gradient are important. Externally produced variations in the

rate of heat flow can result in these perturbations. Other possibilities such as unsymmetric end effects may also be causing bands. Experiments, aimed at eliminating the sources of banding, are being made to evaluate the effects of drafts on the solidification apparatus, end cooling of the ingot, and solidification of longer ingots.

MECHANICAL TESTING PROCEDURE

Tensile tests of several unidirectionally solidified Cu-Cr and Al-Al_3Ni heats and Cr whiskers have been made.

Cu-Cr System

Standard 0.250-in. dia, 1-in. gage length tensile specimens were machined from ingots grown at rates of 10 cm/hr and tested on a Tinius-Olsen four-screw testing machine at a constant strain rate of 0.01 in./in./min using a 1-in. nonaveraging extensometer to measure strain.

Al-Al_3Ni System

Standard 0.250-in. dia, 1-in. gage length tensile specimens were machined from two as-cast and seven unidirectionally solidified (1 to 3 cm/hr growth rates) ingots. These were also tensile tested on a Tinius-Olsen four-screw testing machine at a constant strain rate of 0.01 in./in./min using a 1-in. nonaveraging extensometer to measure strain.

Cr Whiskers

Chromium whiskers were chemically extracted from Cu-Cr eutectic ingots unidirectionally solidified at rates of 2.13 and 3.45 cm/hr using dilute nitric acid. Individual filaments were fastened to tapered 1-mil tungsten jaws with diphenyl carbazide glue, axially aligned and dead-weight loaded to fracture. Strain was measured by taking high-magnification photographs of the filament at various load increments. The tensile testing instrument used to obtain the mechanical properties is described in detail in Ref. 1.

The fractured chromium whisker stub was examined in the electron microscope to determine its apparent thickness and mode of fracture. Selected area electron

diffraction photographs were taken at the fractured tip and along the length of the stub in order to obtain its crystallographic fiber axis.

Sections (1/2 in. x 9/16 in.) cut perpendicular to the growth axis in the equilibrium zone of the Cu-Cr ingots were replicated and electron photomicrographs taken in order to determine a whisker shape factor and mean whisker spacing, and density. These sections were subsequently dissolved to obtain the chromium whiskers for the tensile testing described above.

RESULTS AND DISCUSSION

Preliminary Microstructure Studies of Cr-$Cr_{23}C_6$ System

Microstructural examinations of the as-cast ingot have indicated that the eutectic in this system is rods or whiskers of Cr in a $Cr_{23}C_6$ matrix. This was substantiated by using a concentrated HCl etch to preferentially etch Cr and also by measuring the microhardness of the eutectic matrix and proeutectic Cr islands present in the structure. These microhardness measurements indicated that the eutectic matrix is approximately two times as hard as the proeutectic Cr. These results are contradictory to those first reported by Westbrook (Ref. 2) but are consistent with data subsequently reported by Westbrook in Ref. 3. Since the eutectic structure is not as anticipated ($Cr_{23}C_6$ rods in a Cr matrix) and since such a structure does not meet the fundamental requirements for a reinforced composite structure, no further work will be done on this system. Instead, as previously described, the Al-Al_3Ni system has been chosen as an alternate.

Controlled Solidification Experiments

Cu-Cr and Al-Al_3Ni Systems

The unidirectional solidification experiments on the Cu-Cr and Al-Al_3Ni rod-type eutectic systems have been moderately successful with the exception of occasional banding in the Al-Al_3Ni system. The effect of banding on the mechanical properties in this system is discussed in detail below and it is felt that no major problems arise because of the banding. The specimens have shown only occasional defects of this type which may be located by microstructural examination.

Al-Cu System

Considerable progress has been made toward producing large grains of controlled Al-$CuAl_2$ eutectic, Fig. 1. However, production of band-free ingots from which micro-

bend specimens can be machined has not been accomplished to date. Several heats with very light banding have been produced and, by proper control of the external variables which might cause perturbations, it is felt that ingots sufficiently free of bands can be produced.

Mechanical Test Results

Cu-Cr System

Previous work reported by Hertzberg and Kraft (Ref. 4) indicated that the failure mode of specimens unidirectionally solidified at slow rates (less than 3 cm/hr) was of the shear type. This mode was believed due to a local strain concentration within a narrow necked band 45 degrees to the tensile axis. It was postulated that Cr fibers fractured in this band and formed microvoids which eventually coalesced to produce a "dimpled" fracture surface with Cr fibers being found at the apex of the "elongated dimples." The schematic representation of this proposed fracture mechanism is illustrated in Fig. 4. It should be noted that no increase in strength over that of the tensile strength of Cu was observed for this oriented structure. This was due to the low volume percent (roughly 2 percent) of the ultrahigh strength Cr fibers.

The first task of this program was to study the change in fracture mode, if any, of specimens solidified at faster rates (approximately 10 cm/hr). Several tensile bars were tested to failure and again showed no increase in strength over that of the Cu matrix. The fracture originated within a necked band perpendicular to the tensile direction and produced a jagged fracture surface oriented between 60-90 degrees to the tensile axis. On the basis of preliminary fractographic analysis, the fracture surface appeared to be formed by both a shear and tension mechanism. The shear mechanism is the same as reported above for specimens solidified at the slower rates and is illustrated in Fig. 5. The tension mechanism produces a fibrous structure of highly distorted material with a small number of equiaxed dimples (Fig. 6). This is caused by the surfaces pulling apart rather than by shearing. The Cr fibers do not appear to contribute a major role in this type of separation.

Al-Al_3Ni System

Tensile specimens were prepared from both as-cast and controlled microstructures. Two as-cast samples consisting of an aluminum matrix hardened by 11 volume percent of a randomly dispersed second phase of Al_3Ni rods exhibited an average tensile strength of 13,600 psi and a total elongation of from 15 to 20 percent. Three uni-

directionally solidified test bars, composed of an Al matrix reinforced by Al_3Ni rods aligned parallel to the growth direction, displayed an average tensile strength of 35,000 psi with approximately 2% elongation. Preliminary comparison of tensile properties between the as-cast and the unidirectional solidified specimens is illustrated in Fig. 7. These results indicate that the Al matrix is satisfactorily transferring the major portion of the load to the presumed high-strength Al_3Ni fibers.

The tensile data from these samples is currently being analyzed to determine whether the experimentally obtained maximum strength and general stress-strain behavior may be predicted from theoretical considerations. Preliminary results show that the material does exhibit many of the theoretically predicted characteristics. This analysis will be completed shortly and shall be presented in the next progress report.

When the fibers carry the major portion of the load, failure of the composite should occur a very short time after fiber failure. During the time interval between fiber and composite failure, small voids could form at the fracture site of the fiber. Depending on whether the fracture plane was normal to or at some angle to the fiber axes, the fracture surface should consist either of "equiaxed dimples" containing the outline of the broken Al_3Ni fibers, or "elongated dimples" containing the outline of the broken Al_3Ni fibers, respectively. This mechanism of failure has been verified by means of an electron fractographic study of the fractured surface (Figs. 8 and 9). The photograph of the shear-type failure was taken of a specimen that contained short plates as well as rods so that both shapes of the Al_3Ni phase are noted on the surface.

Two experimental problems have been encountered in the production and testing of this material. Due to some perturbation in the solidification process, some samples have been produced with one or more narrow annular bands perpendicular to the growth direction which are depleted of the Al_3Ni fibers. Consequently, less than optimum tensile properties have been obtained with such a microstructure. The strengths of these test bars were found to be in the range of 25,000 - 30,000 psi with failure always occurring through the band or at the band-normal matrix interface (Fig. 10). These relatively high strengths are produced by the plastic constraint induced triaxial tensile stresses in the Al band.

Failures of the band-free structure have occurred in that shoulder section of the test bar which is nearer to the head end of the original ingot. An effort is currently being made to determine whether these failures are due to the stress concentration in the fillet or to some microstructural inhomogeneity peculiar to the head end of all ingots.

The results obtained to date are most encouraging in that they verify the contention that the binary eutectic alloys can serve as an excellent medium for the study of reinforced structures. The major advantage of this class of materials is that there are no bonding problems associated with the two phases since they are simultaneously produced from the melt.

Cr Whiskers

The mechanical properties of chromium whiskers tested in this report period are shown in Table I. They represent only those whiskers axially aligned and whose fracture occurred within the outermost fiducial beads, with the exception of specimen No. 102. The cross-sectional area of the stub used in determining the tensile stress was assumed to be circular. The corresponding diameter was found by measuring the shadow thickness of the stub when magnified to 12,000 diameters in the electron microscope.

Because of the approximations made in determining the cross-sectional area, precise values of the elastic modulus are difficult to obtain. However, experimentally determined values of the elastic modulus of chromium are within the limits of those calculated from empirically and experimentally obtained compliance constants and crystallographic relationships. For example, the calculated modulus of Cr in the $\langle 112 \rangle$ direction is 40.8×10^6 psi and agrees well with specimens No. 151 and No. 101, whose experimental moduli vary from $34.8 - 40.0 \times 10^6$ psi and whose fiber axis has been determined to be $\langle 112 \rangle$.

When the elastic limit was exceeded at room temperature, the whiskers either fractured with no measurable deviation from Hooke's law (Fig. 11) or displayed some nonlinear behavior (Fig. 12). Prolonged time at constant load produced no measurable creep.

Two specimens, Nos. 101 and 102, fractured in a spectacular manner. They snapped suddenly and kinked around themselves. Upon detaching the whisker from the tensile jaws by heating the glue to 285 C, the kink immediately straightened out. Other specimens, not reported here, fractured so violently that the stubs were lost and no area measurements or failure mode observations could be made.

These tensile measurements are being made on Cr whiskers which have a thin passive surface film as a consequence of being stored in air for a minimum of several days. This procedure must be followed since freshly extracted whiskers will burn when heated to the mounting temperature. It should be noted that X-ray diffraction data do not indicate the presence of a chromium oxide film on whiskers which have been stored in air for over one year.

FUTURE WORK

During the next report period, continued attempts will be made to produce band-free unidirectionally solidified ingots of the Al-$CuAl_2$ eutectic with large grains from which microbend specimens may be machined. These specimens will be oriented in such a way that the tensile properties and elastic moduli may be determined as a function of lamellae orientation.

Similarly, band-free specimens of the Al-Al_3Ni will be tested and the cause of the shoulder failure in those band-free specimens reported above will be assessed. Work will continue toward predicting the mechanical behavior of the controlled composite structure using first principles of elastic theory and an extension of these principles to predict the behavior after plastic deformation of the matrix will be attempted. At the same time, the feasibility of chemically separating the Al_3Ni rods (whiskers) from the Al matrix and tensile testing them will be assessed.

Work will continue toward obtaining a full understanding of the mode of failure associated with the structure produced at high solidification rates in the Cu-Cr system. Sub-size tensile specimens will be prepared to study the effect of Cr whisker orientation on the failure mode in this system. The first set of specimens will be prepared such that the Cr whiskers are initially at 45 degrees to the tensile axis.

Since the behavior of the Cr whiskers varies from one whisker to another it is difficult to draw conclusions from single tests. Therefore, a number of data points will be obtained for several solidification rates and a statistical analysis of these data attempted.

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3. Westbrook, J.: Private communication, February 1963.

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TABLE I

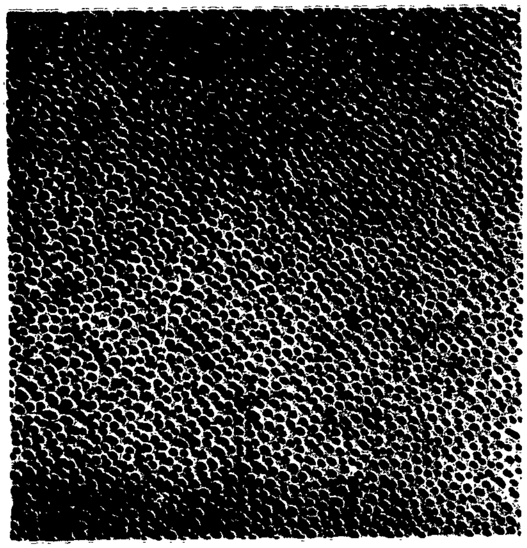
Summary of Results of Tensile Testing of Extracted Chromium Whiskers

Specimen No.	Diameter μ	Gauge Length mm	Maximum Elastic Strain %	Fracture Strength psi	Modulus of Elasticity psi	Fiber Axis	Mode of Fracture
101	0.34	0.50	3.42	$1,290 \times 10^3$	34.8×10^6	[112]	Ductile, kinked
102	0.28	0.38	< 5.70* 3.76 (corrected)	$1,340 \times 10^3$	$> 22.9 \times 10^6$* 36.0×10^6 (corrected)	Unknown	Kinked
105	0.35	0.56	2.27	$1,150 \times 10^3$	45.7×10^6	Unknown	Shear
107	0.60	0.49	1.68	620×10^3	34.0×10^6	Unknown	Unknown
151	0.45	0.53	1.93	$1,110 \times 10^3$	40.0×10^6	[112]	Ductile, kinked
154	0.57	0.63	2.10	969×10^3	37.5×10^6	Unknown	Shear
155	0.82	0.70	1.68	585×10^3	34.0×10^6	Unknown	Shear

* Measured strain in error due to slippage of filament in glue at jaws. Strain is corrected on basis of bulk modulus of Cr.

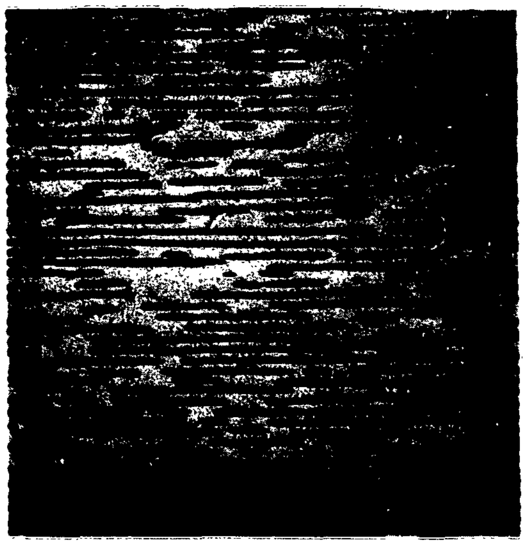
MICROSTRUCTURE OF UNIDIRECTIONALLY SOLIDIFIED Al-Al_3Ni EUTECTIC SPECIMEN

a) TRANSVERSE SECTION; GROWTH DIRECTION NORMAL TO PAGE
MAGNIFICATION: 500 X



62-625-02

b) LONGITUDINAL SECTION; GROWTH DIRECTION LEFT TO RIGHT
MAGNIFICATION: 200 X



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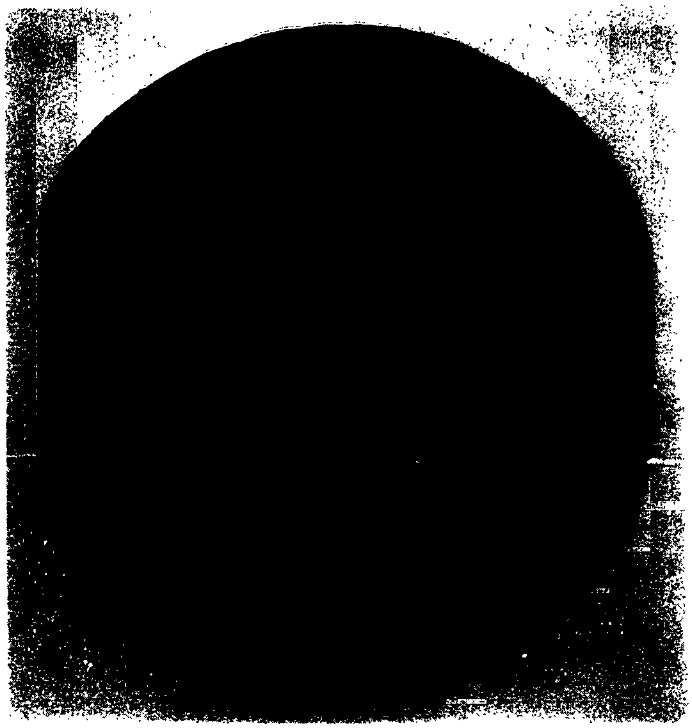
MACROSTRUCTURE OF Cu-Al UNIDIRECTIONALLY SOLIDIFIED EUTECTIC ALLOY

TRANSVERSE SECTION
MAGNIFICATION: 3 X



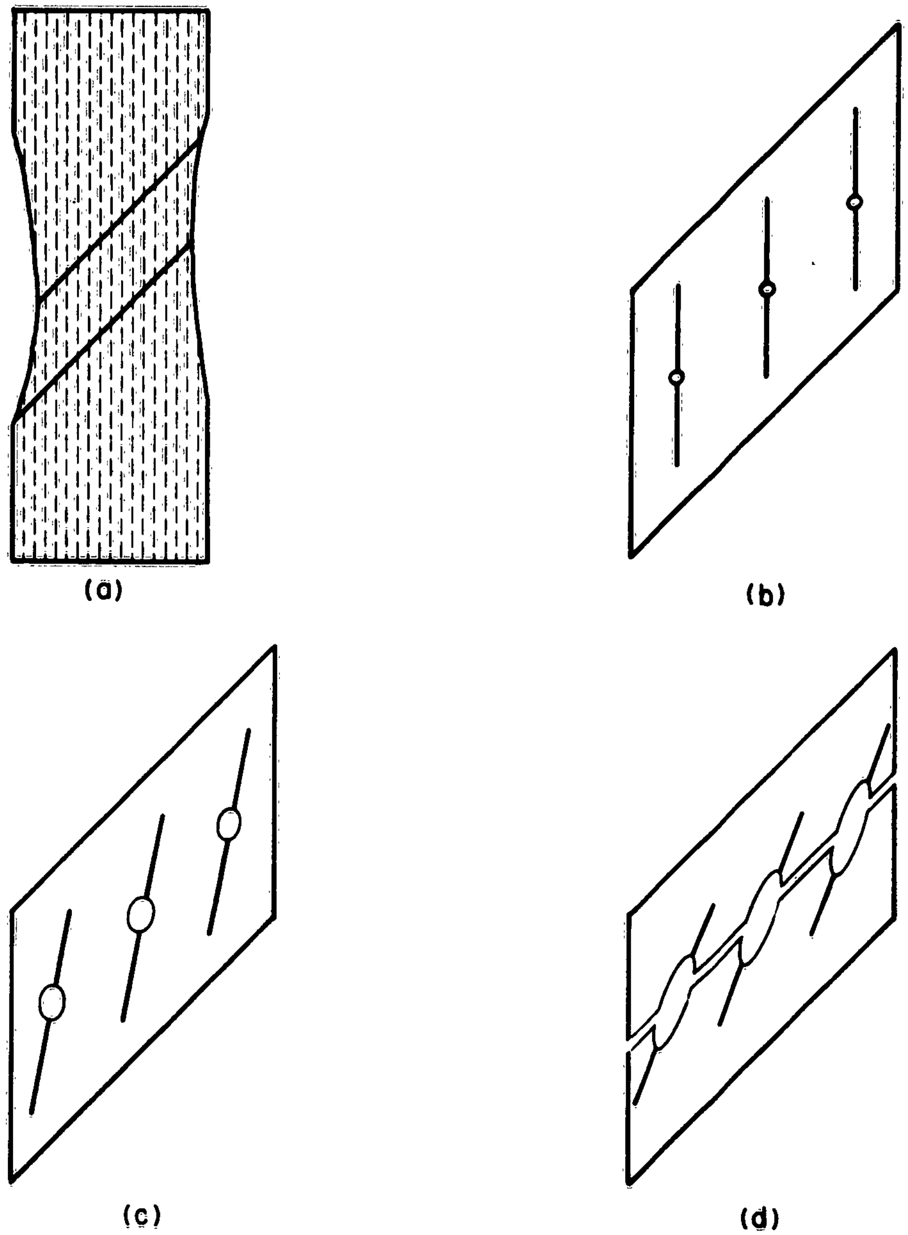
63-105-02-01

MACROSTRUCTURE OF Cu-Al UNIDIRECTIONALLY SOLIDIFIED EUTECTIC ALLOY

LONGITUDINAL SECTION INDICATING SEVERE BANDING
SPECIMEN IS OVERETCHED
MAGNIFICATION: 3X

63-069-01-01

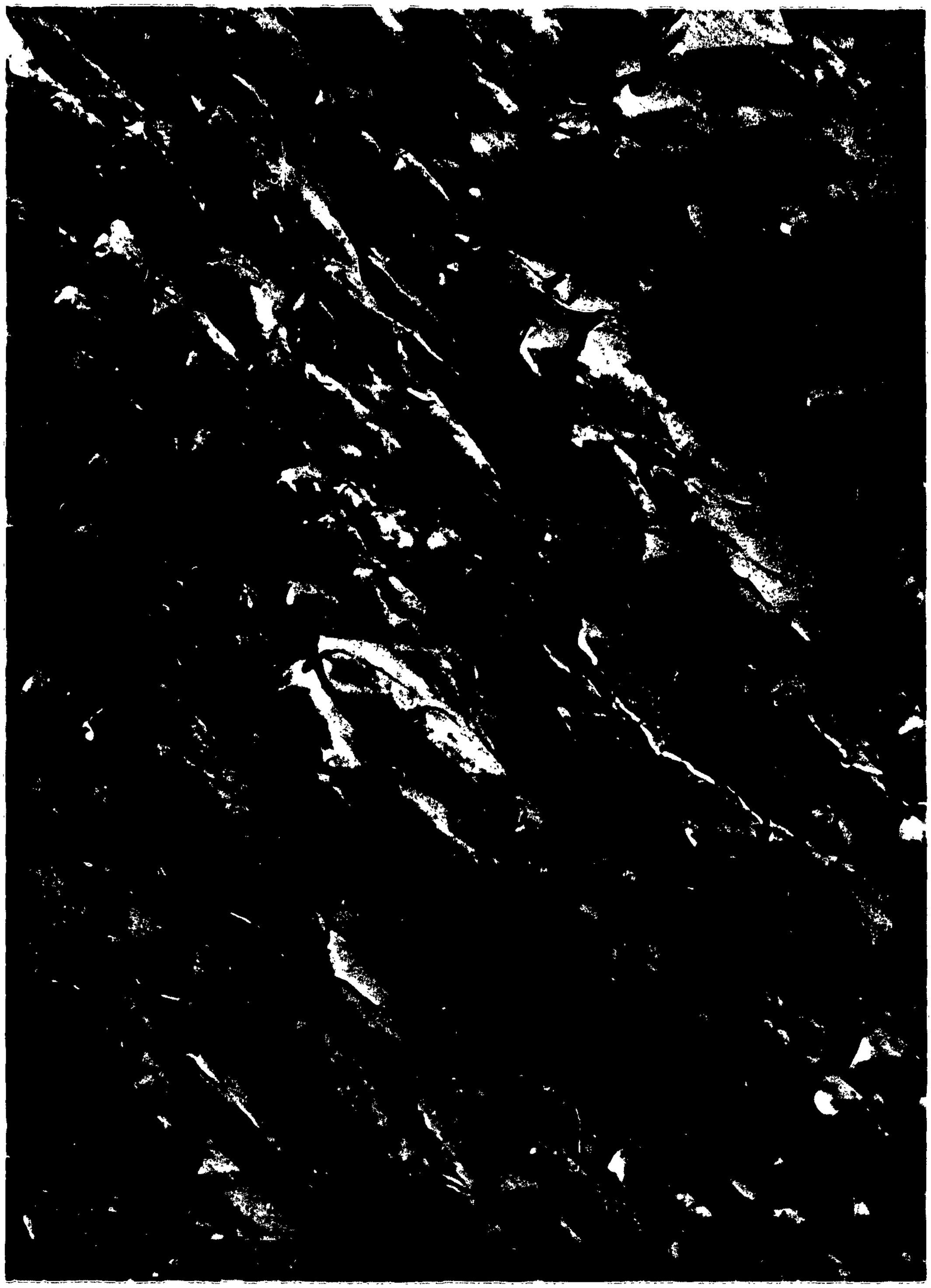
SCHEMATIC REPRESENTATION OF PROPOSED SHEAR FRACTURE MODE IN CONTROLLED Cu-Cr ALLOY



(a) SHEAR BAND IN NECKED REGION
(b) WHISKERS FRACTURE CREATING VOIDS
(c) DEFORMATION OF MATRIX ROTATES WHISKERS
(d) FRACTURE ALONG SHEAR BAND CONNECTS VOIDS PRODUCING "ELONGATED DIMPLES" WITH OUTLINES OF WHISKERS

"ELONGATED DIMPLES" ASSOCIATED WITH SHEAR MODE OF FAILURE

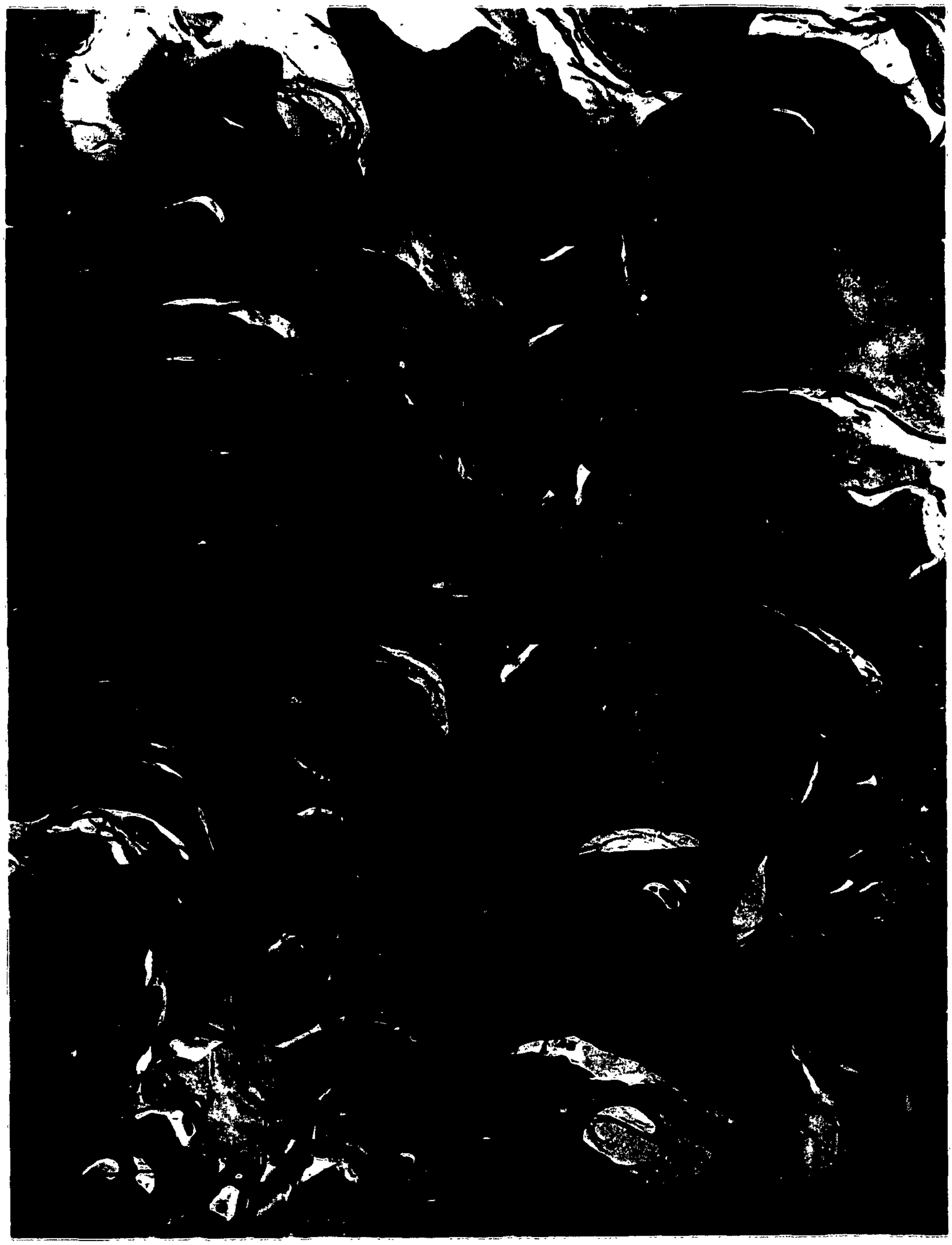
MAGNIFICATION: 13500 X



E63-042 62-604

FIBROUS STRUCTURE ILLUSTRATING TENSILE MODE OF FAILURE

MAGNIFICATION: 14200 X



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COMPARISON OF TENSILE PROPERTIES OF AS-CAST AND UNIDIRECTIONALLY SOLIDIFIED Al-Al_3Ni EUTECTIC ALLOY

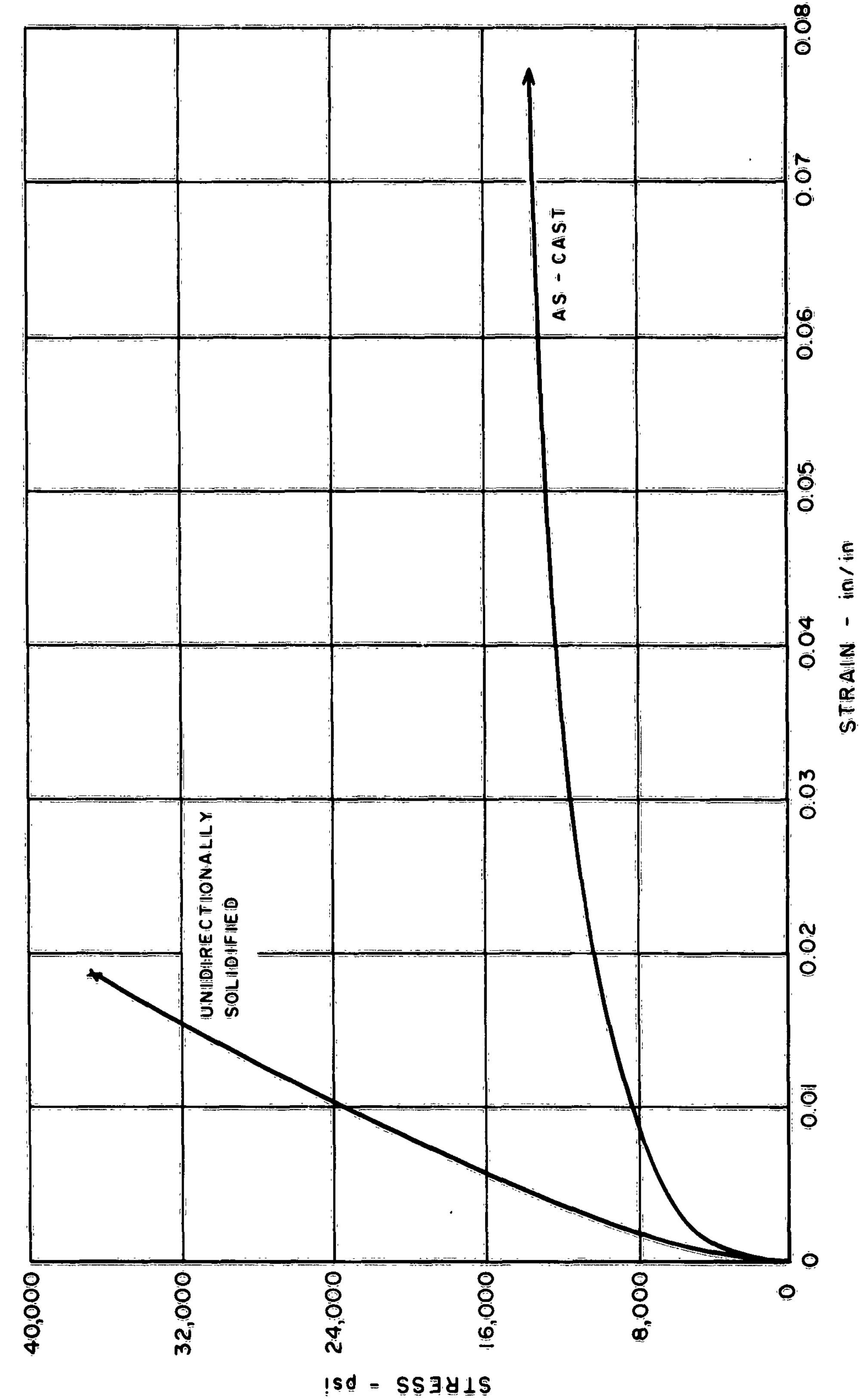

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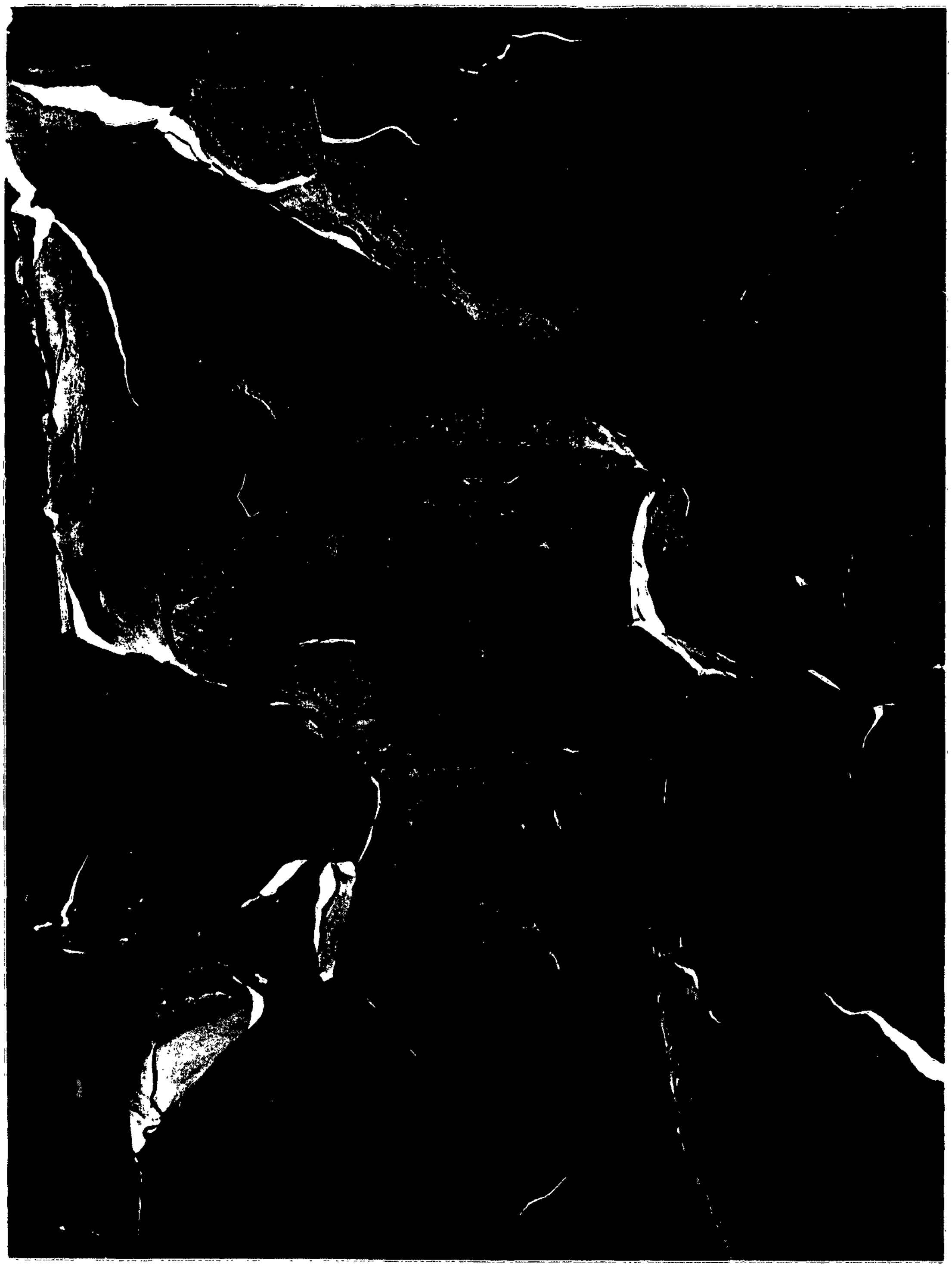
FIG. 8

FRACTURE SURFACE OF Al-Al_3Ni EUTECTIC ALLOY

"EQUIAXED DIMPLES" INITIATED BY FAILURE OF Al_3Ni FIBERS IN PLANE NORMAL TO TENSILE AXIS

MAGNIFICATION: 8300 X



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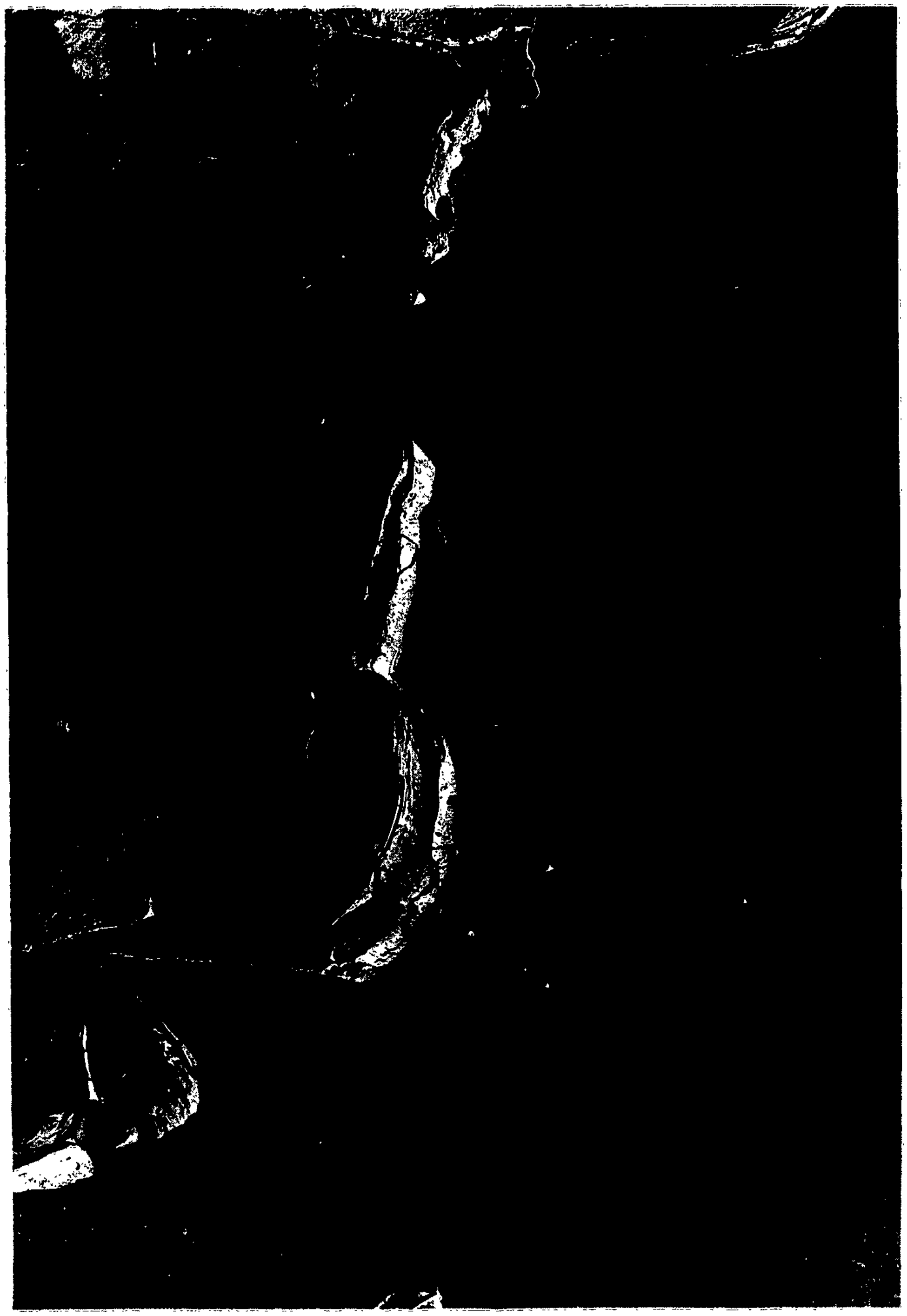
FRACTURE SURFACE OF Al-Al_3Ni EUTECTIC ALLOY

"ELONGATED DIMPLES" INITIATED BY FAILURE OF Al_3Ni PLATES AND RODS IN PLANE AT ANGLE TO TENSILE AXIS

MAGNIFICATION: 7100 X



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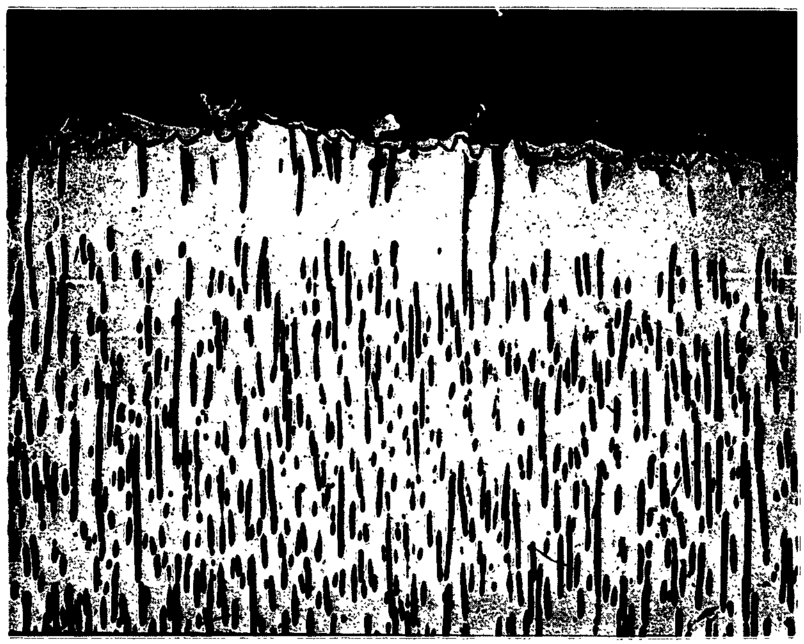
FIBER - FREE BAND ADJACENT TO FRACTURE SURFACE

BAND CAUSED BY PERTURBATION DURING SOLIDIFICATION.
FRACTURE SURFACE NICKEL PLATED TO PREVENT
ROUNDING DURING POLISHING

MAGNIFICATION: 100 X



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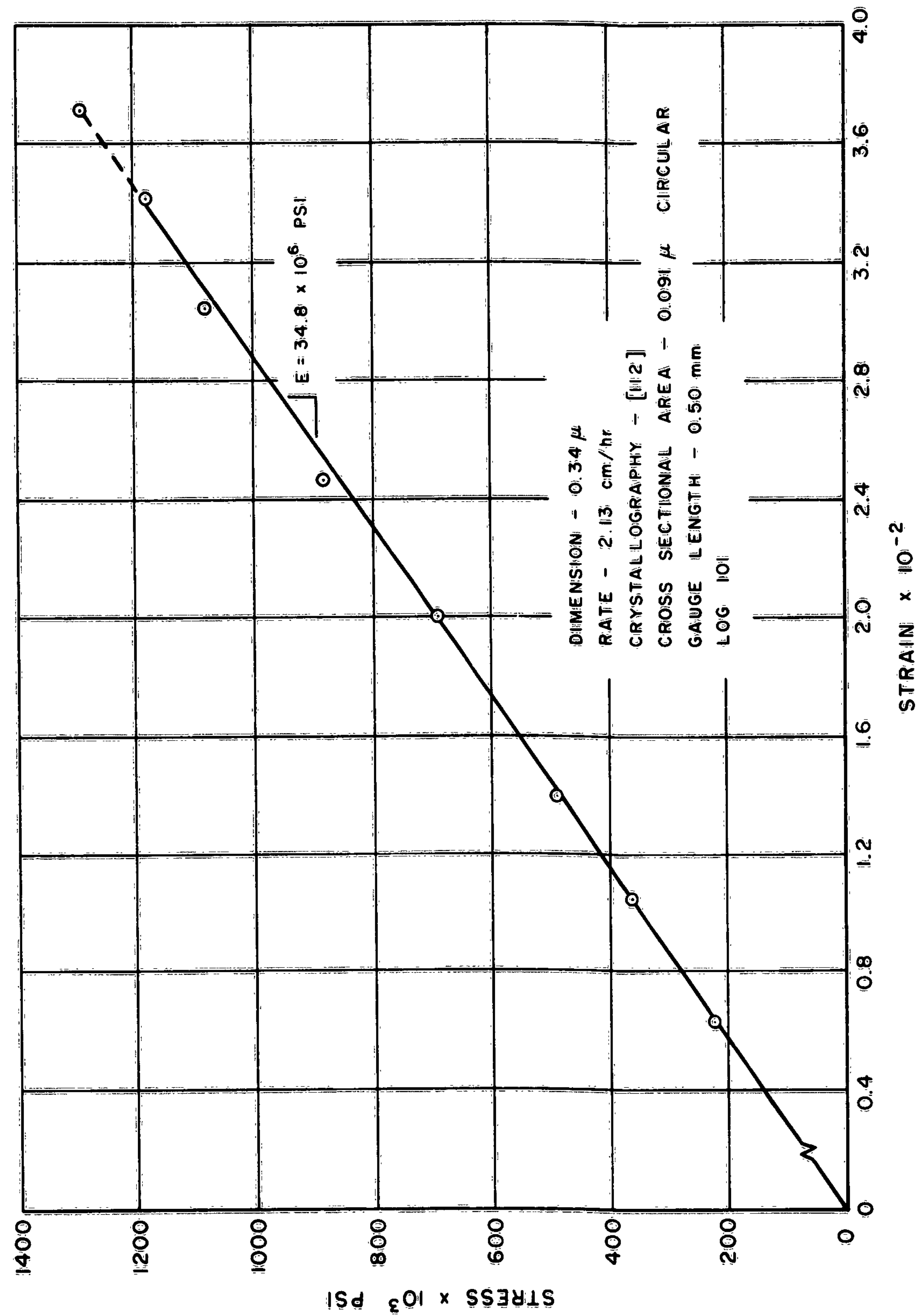
STRESS - STRAIN CURVE FOR Cr WHISKER



STRESS - STRAIN CURVE FOR Cr WHISKER